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BISPHENOL-A PRODUCTION METHOD  
[Bisufenouru no seizouhouhou]

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## Specification

### 1. Name of this Invention

BISPHENOL-A PRODUCTION METHOD

### 2. Claims

[1] Bisphenol-A production method comprising a step of obtaining a reaction product by reacting phenol and acetone in the presence of acid catalyst, step of obtaining a liquid mixture by removing an acid catalyst from said reaction product, and step of treating said liquid mixture with a weakly basic ion-exchange resin having pyridyl group as exchange group.

### 3. [Detailed Explanation of this Invention]

#### [Field of the Invention]

The present invention relates to a bisphenol-A production method. In detail, the method of this invention obtains a liquid mixture by removing an acid catalyst from a reaction product and treats said liquid mixture with a weakly basic ion-exchange resin having pyridyl group as exchange group, thus producing bisphenol-A having high purity.

An growing demand exists for bisphenol-A, which is useful as a raw material for polycarbonate resin, epoxy resin and engineering plastics, etc.

For those purposes, bisphenol-A must be colorless and highly purified.

[Prior Art]

Bisphenol-A is produced by reacting phenol and acetone in the presence of acid catalyst.

In addition to bisphenol-A, the reaction product contains byproducts of catalyst, non-reacted acetone, phenol, water, colored substance, or the like.

Examples of catalyst used for this condensation reaction are an inorganic acid such as hydrochloric acid and weakly acid positive ion-exchange resin.

Numerous methods are known for producing highly purified bisphenol-A from said reaction product.

When hydrochloric acid is used as a catalyst, the reaction product is heated to 110°C - 120°C under reduced pressure to remove hydrochloric acid, non-reacted acetone, water and a small amount of phenol so as to separate bisphenol-A and phenol additive. Another method is that, to separate high boiling point bisphenol-A from low boiling point bisphenol-A, the reaction product is distilled, and in some cases, the product is extracted or recrystallized with a solvent.

When hydrochloric acid is used as a catalyst, a trace of hydrochloric acid remains after distillation to cause various difficult problems to the succeeding production processes.

One reason is that the production device is corroded to create metallic salt, thereby producing impure bisphenol-A. Also, a large

scale of refinement is necessary.

If the devices are made highly resistant to acid, the device cost will be too high.

Another problem is that, as described in Pat. No. 38-4875, the acid substance produced by distillation decomposes bisphenol-A.

When a weak acid positive ion-exchange resin is used as a catalyst, separation of catalyst is easier than separation of hydrochloric acid catalyst. However, if it is within the reaction range of 70°C - 100°C, a trace of free acid remains due to the separation of exchange group to cause the same problem as experienced with hydrochloric acid catalyst.

Pat. No. 49-1543 disclosed that, if bisphenol-A and bisphenol additive are melted and processed with an acid and/or weakly basic ion-exchange body, bisphenol-A can be distilled without decomposing bisphenol-A.

However, to melt bisphenol-A and phenol additive, the temperature higher than 90°C is required.

The problem with this method is that, for a long duration of reaction, the upper limit of the temperature for weakly basic ion-exchange resin having 2-class or 3-class amine as an exchange group is usually 70°C - 80°C. If said weakly basic ion-exchange resin is used at the temperature higher than this range, exchange groups separate to color the processing liquid to yellow - red. Therefore, weakly basic ion-exchange resin cannot be used.

#### [Problems to Be Solved by this Invention]

The object of this invention is to provide a bisphenol-A production method that can prevent the problems experienced with the conventional method (i.e., corrosion of device by a small amount of acid; decomposition of bisphenol-A during distillation).

#### [Method and Operation for Solving the Problems]

The developers of this invention investigated a method to solve the problems described above and discovered that, after removing an acid catalyst from the reaction product, by treating the product with a specific weakly basic ion exchange-resin, the object of this invention could be achieved.

That is, this invention provides a bisphenol-A production method comprising a step of obtaining a reaction product by reacting phenol and acetone in the presence of acid catalyst, step of obtaining a liquid mixture by removing an acid catalyst from said reaction product, and step of treating said liquid mixture with a weakly basic ion-exchange resin having pyridyl group as exchange group.

The molar ratio of phenol and acetone is normally 4 - 12, and reaction is performed at 40°C - 100°C.

As an acid catalyst, evaporative acid, such as hydrochloric acid, or insoluble acid, such as strongly acid positive ion-exchange resin, may be used.

When hydrochloric acid is used, the reaction product flowing out from the reaction band area forms a liquid mixture, as water,

hydrochloric acid and a small amount of phenol are removed by distillation under reduced pressure. The distillation is preferably conducted under 20 - 200 mmHg pressure at 90 - 150°C.

Also, when a strong acid positive ion-exchange resin is used, water, hydrochloric acid and a small amount of phenol are removed by distillation under reduced pressure to form a liquid mixture. The distillation is preferably conducted under 50 - 300 mmHg pressure at 70 - 120°C.

Examples of weakly basic ion-exchange resin having a pyridyl group as an exchange group are a copolymer of 2-vinyl pyridine and/or 4-vinyl pyridine and divinyl benzene, copolymer of 2-vinyl pyridine and/or 4-vinyl pyridine and divinyl benzene and styrene, or the like.

As the upper limit of reaction temperature of those resin is high, the operational temperature is usually 150°C or below.

The acid density of said liquid mixture supplied to this resin should be 0 - 2 equivalent portion/TON, preferably 0 - 1 equivalent portion/TON.

The treating method may be continuous or batched, and the temperature is preferably at 70 - 150°C.

When a continuous method is used, the flow of liquid mixture should be 10 - 1,000 Kg/Hr per 1 Kg of resin.

If a batch method is selected, 1 - 20 wt% of resin are preferably used for the liquid mixture for 5 - 60 min.

After the treatment with resin, highly purified bisphenol-A can be obtained.

That is, by cooling the processing liquid, bisphenol-A is crystallized as an additive material with phenol. Then, this crystal is separated, and phenol is removed from the additive product to collect bisphenol-A. Another method is that, after phenol is removed from the processed liquid, bisphenol-A is distilled and recrystallized with a solvent.

#### [Operational Examples]

The following describes the operational examples of this invention.

Note that % in the examples designates weight %.

#### Operational example 1:

564 g of phenol and 58 g of acetone were condensed in the presence of hydrochloric acid catalyst. Then, hydrochloric acid, water, and a small amount of phenol were removed from the reaction product under the final pressure of 70 mmHg at 120°C.

The obtained hydrochloric density in the dehydrochlorinated crystal was 0.25 equivalent portion/TON.

A weakly basic ion-exchange resin having 5% of pyridyl Group as exchange group (Kosei Kagaku Kogyo, KEX 212) was mixed in this dehydrochlorinated crystal. Then, after the mixture was stirred at 120°C for 15 min, the resin was separated by filtration.

Hydrochloric acid was not detected in the liquid.

Next, phenol was removed from this liquid under the final pressure of 10 mmHg at 170°C, and bisphenol-A was distilled. As a result, as decomposition of bisphenol-A did not occur, white bisphenol-A was obtained.

#### Comparison example 1:

The same method as described in the Operational example 1 was performed, except that KEX-212 was not used.

The hydrochloric acid density in the dehydrochlorinated crystal was 0.27 equivalent portion/TON.

Bisphenol-A was decomposed while being distilled. The distilled bisphenol-A was colored in yellow.

#### Operational example 2:

The same method described in the Operational example 1 was performed from the reaction to the process using KEX-212.

The obtained hydrochloric density in the dehydrochlorinated crystal was 0.27 equivalent portion/TON. However, after processing with KEX-212, hydrochloric acid was not detected.

Next, 3% water was added to this processed liquid and stirred at 120°C for 48 hours in a nitrogen atmospheric stainless container. Then, the liquid was cooled to 45°C to crystallize phenol and bisphenol-A additive.

This additive was separated by filtration and washed with an equal amount of phenol.

When the obtained additive (20 g) was dissolved in 20 ml of ethanol, the Hazen color was 5 APHA.

Comparison example 2:

The same method as described in the Operational example 2 was performed, except that KEX-212 was not used.

The hydrochloric acid density in the dehydrochlorinated crystal was 0.20 equivalent portion/TON.

Although stainless corrosion was not visible, when 20 g of obtained additive were dissolved in 20 ml of methanol, the Hazen color was 30 APHA.

Comparison example 3:

The same method as described in the Operational example 2 was performed, except that KEX-212 was replaced with a weakly basic ion-exchange resin having 3-class amine as exchange group (Byer, Levachit MP-62).

The hydrochloric acid density in the dehydrochlorinated crystal was 0.20 equivalent portion/TON. The hydrochloric acid was not detected after the treatment with MP0-62. However, the liquid was colored in red.

When 20 g of obtained additive were dissolved in 20 ml of methanol, the Hazen color was 50 APHA.

[Effectiveness of this Invention]

As described above, the present invention can prevent decomposition of bisphenol-A at high temperature.

Furthermore, the method of this invention can prevent bisphenol-A from becoming impure by device corrosion. Therefore, the obtained bisphenol-A can present excellent color phase.